Powder Metallurgical Processing of Aluminium Metal Matrix Composites Reinforced with Graphite

by

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CERTIFICATION OF APPROVAL

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Mechanical Engineering Programme
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Approved by,

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September 2011

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NURUL AIZAD BINTI MD SAFIAN

ABSTRACT

In aircraft and automotive industries, it is preferable to use lightweight yet strong material especially aluminium. Due to high strength to weight ratio, automobile and aircraft component are generally made out of Aluminium alloy which makes the transport lighter and indirectly results in saving fuel consumption. However, the used of Aluminium alloy has been limited because of unfavourable wear. In this project, to improve the wear resistance, the author has dispersed graphite particles in the aluminium metal matrix composites. Aluminium-Graphite composite was processed and was used for the analysis. The main objective of this project is to further reduce the density of aluminium matrix reinforced with graphite. Other than that, this project also done to investigate the wear resistance behaviour of the composites with different amount of graphite. The third objective of this project is to compare the microstructure and physical properties between aluminium and aluminium reinforce with graphite with different percentage of graphite amount. All the samples needed in this research were prepared by the author using powder metallurgy hot pressing process. The wear behaviour of the composites with different amount of graphite was investigated using pin on disc method while the hardness of each sample with different weight percentage of graphite was determined using Vickers microhardness test machine. Then, the author examines the improvement of sample's microstructure using Scanning Electron Micrograph (SEM) and X-Ray Diffraction (XRD) method. The result shows that the density of the composites is decreased after hot pressing process. Hardness of the unreinforced aluminium matrix is improved from 22.2HV to 66.7HV after been reinforced with increasing wt% of graphite. From the experiment, adding only 2% of graphite results in highest wear resistance. As for the micrograph analysis, the increment of graphite amount in aluminium based composites is proven and no new phases are formed in the composites. Most of the objectives of this project are achieved except for wear resistance behaviour where the results obtained are not as expected by the author but still it is a good finding.

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CHAPTER 1

INTRODUCTION

1.1 Background

Composite materials are engineered materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure [1].

Over the last three decades, another class of material, namely the metal matrix composites (MMCs), is becoming increasingly important and drawing attention of engineers. MMCs normally fabricated using ductile metal such as aluminium, titanium or nickel as the matrix material and reinforcement usually comprises particles of a ceramic second phase such as silicon carbide, alumina, graphite, etc.

This class of material has been widely studied by numerous researchers with respect to friction and wear behavior. Discontinuously reinforced aluminium matrix composites have emerged from the need for light weight, high stiffness materials which are desirable in many applications such as high speed reciprocating machinery. Significant increase in stiffness and strength can be consulted with even small reinforcement volume fractions. Many of the applications for which MMCs are desirable also require improved tribological performance.

The physical and mechanical properties that can be obtained with metal matrix composites (MMCs) have made them attractive candidate materials for aerospace, automotive and numerous other applications. More recently, particulate reinforced MMCs have attracted considerable attention as a result of their relatively low costs and characteristic isotropic properties.

However, the usages of metal matrix composites are still at the beginning of the evolution curve of modern materials.

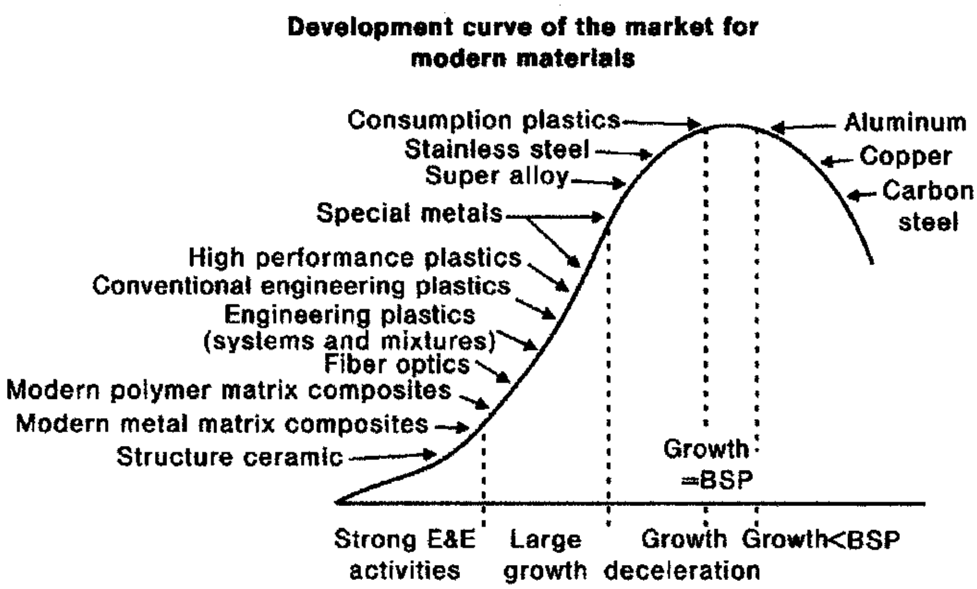


Figure 1.1: Development curve of the market for modern materials [2].

Increased demand for lightweight components, mostly driven by the need to reduce energy consumption in a diversity of communal and structural components, has led to increased utilize of aluminium. Internationally, the pattern of consumption is more favorable to transportation, mainly due to large-scale aluminium utilization by the aviation space. Besides, with the automotive sector expected to continue its growth to around 900 000 vehicles a year by 2010, there is every reason to expect largely increase in demand for lightweight material. Furthermore, the cost of fabrication together with a need to develop part recovery has led to numerous growths in the net shaped component manufacturing processes.

Powder metallurgical processing of aluminium matrix reinforced with graphite offers components with excellent mechanical and fatigue properties, low density corrosion resistance, high thermal and electrical conductivity, excellent reaction to a range of finishing processes, and which are competitive on a cost per unit volume basis. Besides, it also can be further processed to exterminate porosity and improve bonding yielding properties that compare favourably to those of conventional wrought aluminium product.

1.2 Problem Statement

Due to high strength to weight ratio, automobile and aircraft component are usually made out of lightweight yet strong material especially aluminium. Aluminium alloy will makes the transport lighter and indirectly results in saving fuel consumption. However, the used of aluminium alloy has been limited because of unfavourable wear. In this project, to improve the wear resistance, it has been proposed to disperse graphite particles in the aluminium metal matrix composites. It is predicted by adding graphite in aluminium alloys would improve friction and wear resistance at the same time reducing its weight to strength ratio.

Other than that, since graphite particle are lighter than aluminium metal matrix composites due to the density of graphite is 2.26 g/cm^3 while the density of aluminium is 2.7 g/cm^3, Aluminium-Graphite are used to further reduce the weight of components. Furthermore, it is expected that graphite particulate will enhance the strength of aluminium composite by making the surface harder than pure aluminium.

1.3 Objective and Scope of Study

The objective of this project is divided into three significance purposes which are:

1. To further reduce the weight of aluminium carbon component.
2. To investigate the improvement of wear performance of aluminium composites.
3. To compare the microstructure and physical properties between aluminium and aluminium reinforce with graphite with different percentage of graphite amount.

The samples of aluminium matrix reinforced with graphite were prepared using powder metallurgy hot press process. The process begins with the mixing of aluminium powder with graphite particle using ball mill machine at different weight fraction addition of graphite. Then the powders were compacted and sintered simultaneously using hot press machine with pressure of 6500lb (45 MPa), temperature of 580°C and duration of 60 minutes. The process was done in vacuum atmosphere. The density of each sample after hot pressing was measured using balance machine.

The wear behavior results were obtained in the form of coefficient of weight loss. The author measure the sliding wear characteristics of each samples using pin on disc apparatus following the standard of ASTM G99 - 05(2010) Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus.

The microstructure of each sample were observed using Scanning Electron Microscope (SEM) which follow the following E 986 – 04 Standard Practice for Scanning Electron Microscope Beam Size Characterization and also, X-Ray Powder Diffraction analysis is done to each samples to investigate the behavior of phase changing and its lattice structure. The hardness of each sample which contains different graphite amount will be tested using Vickers Microhardness test machine follow E 92 – 82 (Reapproved 2003) Standard Test Method for Vickers Hardness of Metallic Materials.

CHAPTER 2

LITERATURE REVIEW

2.1 Metal Powders

There are some reasons to use powder and of course powder compaction. Several materials cannot be used in other manufacturing methods because the powder compaction method is verified to be more efficiently favorable method than other manufacturing methods. One type of material used in powder compactions is materials with a very high melting point which is refractory metals, where casting would not be cost-effective because of the high melting points [3].

Composites materials, which cannot simply be mixed in other manufacturing methods or handled in post operation due to its constituents is the other class of material used in powder compactions. Ferrous metals such as iron based metals, copper, brass, bronze aluminium and titanium are the most commonly material used in powder compaction manufacturing method. All the powder materials mentioned above are fabricated using different ways depends on their mechanical behavior.

2.2 Aluminium (Al)

Aluminium is a silvery white member of the boron group of chemical elements. Physically, chemically and mechanically aluminium is a metal like steel, brass, copper, zinc, lead or titanium. It can be melted, cast, formed and machined much like these metals and it conducts electric current. In fact often the same equipment and fabrication methods are used as for steel.

Aluminium is a very light metal with a specific weight of 2.7 g/cm^3, about a third that of steel. For example, the use of aluminium in vehicles reduces dead-weight and energy consumption while increasing load capacity. Its strength can be adapted to the application required by modifying the composition of its alloys. The physical properties of aluminium is shown in APPENDIX A

Aluminium naturally generates a protective oxide coating and is highly corrosion resistant. Different types of surface treatment such as anodising, painting or lacquering can further improve this property. It is particularly useful for applications where protection and conservation are required. Other than that, aluminium is an excellent heat and electricity conductor and in relation to its weight is almost twice as good a conductor as copper. This has made aluminium the most commonly used material in major power transmission lines.

Aluminium is a good reflector of visible light as well as heat, and that together with its low weight makes it an ideal material for reflectors in, for example, light fittings or rescue blankets. Aluminium is ductile and has a low melting point and density. In a molten condition it can be processed in a number of ways. Its ductility allows products of aluminium to be basically formed close to the end of the product's design.

2.3 Graphite

Carbon has two natural crystalline allotropic forms: graphite and diamond. Each has its own distinct crystal structure and properties. Graphite is generally grayish black, opaque and has glossy black lustre. It is unique because it has properties of both a metal and non metal. It is flexible but not elastic, and has a high thermal and electrical conductivity. It is highly refractory and chemical inert. Graphite holds the difference of being the most stable form of carbon under standard conditions. There are two main types of graphite which are natural and synthetic graphite. The physical property of graphite is shown in APPENDIX A.

2.3.1 Natural Graphite

Natural Graphite is a mineral consisting of graphitic carbon. It varies considerably in crystallinity. Most commercial (natural) graphites are mined and often contain other minerals. It is stable over a wide range of temperatures. Graphite is a highly refractory material with a high melting point (3650°C.).

2.3.2 Synthetic Graphite

Synthetic graphite can be produced from coke and pitch. It tends to be of higher purity though not as crystalline as natural graphite. There are essentially two types of synthetic graphite. The first is electrographite, which is pure carbon produced from calcined petroleum coke and coal tar pitches in an electric furnace. The second type of synthetic graphite is produced by heating calcined petroleum pitch to 2800°C. On the whole synthetic graphite tends to be of a lower density, higher porosity and higher electrical resistance. Its increased porosity makes it unsuitable for refractory applications. Synthetic Graphite consists mainly of graphitic carbon that has been obtained by graphitization, heat treatment of non-graphitic carbon, or by chemical vapor deposition from hydrocarbons at temperatures above 2100K.

2.4 Composites

Composite materials are engineered materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure [1].

It's a combinations of two materials in which one of the materials, called the **reinforcing phase**, is in the form of fibers, sheets, or particles, and is embedded in the other materials called the **matrix phase**. Discontinuous phase; which is the reinforcing material are usually strong with low densities and harder than the continuous phase; which is the matrix that is more ductile and tough. The reinforcing particle fillers are widely used to improve the properties of matrix materials. The combination of strength from the reinforcement with the toughness of the matrix may produce desirable properties which are not available in any single conventional material. The reinforcing material and the matrix material can be metal, ceramic, or polymer.

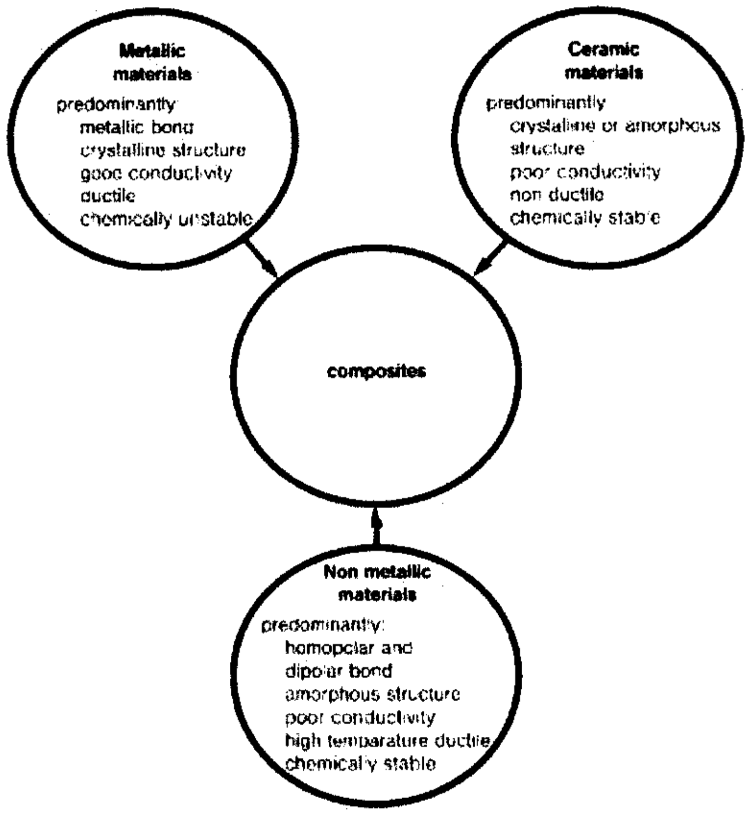


Figure 2.1: Classification of the composites materials within the group of materials [2].

2.5 Metal Matrix Composites

Metal matrix composites (MMCs) are composed of an element or an alloy matrix in which the second phase is embedded and distributed to achieve some property improvement. The reinforcement is called as discontinuous material. The reinforcement materials are typically ceramics which provide a very beneficial combination of stiffness, strength, and relatively low in density. Generally, by increasing the weight fraction of the reinforcement phase in the matrix, stiffness, yield strength and tensile strength, its resistance towards wear will also increase at the same time reducing its weight. Metal matrix composites have outstanding benefits due to the combination of metallic and ceramic properties which contribute to the improvement of its yielding, physical and mechanical properties.

Every reinforcement has a typical profile, which is significant for the effect within the composite material and the resulting profile. Table 1 gives an overview of possible property profiles of various material groups.

Table 2.1: Property potential of different metal matrix composites [2].

MMC type	*Properties Strength*	*Young's modulus*	*High temperature properties*	*Wear*	*Expansion coefficient*	*Costs*
mineral wool: MMC	*	*	**	**	*	medium
discontinuous reinforced MMC	**	**	*	***	**	low
long fiber reinforced MMC: C fibers	**	**	**	*	***	high
other fibers	***	***	***	*	**	high

From table above, we can see that discontinuous reinforced MMC offers the best conditions for reaching development targets.

2.5.1 Aluminium Metal Matrix Composites

In AMCs one of the constituent is aluminium/aluminium alloy, which forms percolating network and is termed as matrix phase [2].

Over the years, AMCs have been tried and used in numerous structural, non-structural and functional applications in different engineering sectors. Driving force for the utilization of AMCs in these sectors include performance, economic and environmental benefits [4]. The advantages of AMCs that makes it favorable in transportation sector are lower fuel consumption, less noise and lower airborne discharges. With increasing severe environmental regulations and highlighting on improved fuel economy, use of AMCs in transport sector will be inescapable and worthy in the coming years.

2.6 Aluminium Metal Matrix Composites Reinforced with Graphite

Aluminium MMCs reinforced with graphite is particulate aluminium matrix composites (PAMCs). The mechanical properties of PAMCs are inferior compared to whisker/short fibre/continuous fibre reinforced AMCs but far superior compared to unreinforced aluminium alloys [4]. For a material to be a reinforcement substance, it must follow such demand as has been stated by Karl Ulrich Kainer et. al (2006) which are [2]:

- Low in density
- High Young's modulus
- High compression and tensile strength
- Lower thermal expansion coefficient
- Low in cost

In this experiment, author use graphite as reinforcement because graphite has low in density, moderate Young's modulus, moderate tensile and compression strength and low thermal conductivity.

The major advantages of PAMCs compared to unreinforced materials are as follows:

- Greater strength
- Improved stiffness
- Reduced density (weight)
- Improved abrasion and wear resistance

Based on a research done by Mohd Shukor bin Salleh from Universiti Teknikal Malaysia Melaka [3], by increasing the weight percentage of reinforcement materials in the AMCs, density will decrease and hardness will increase.

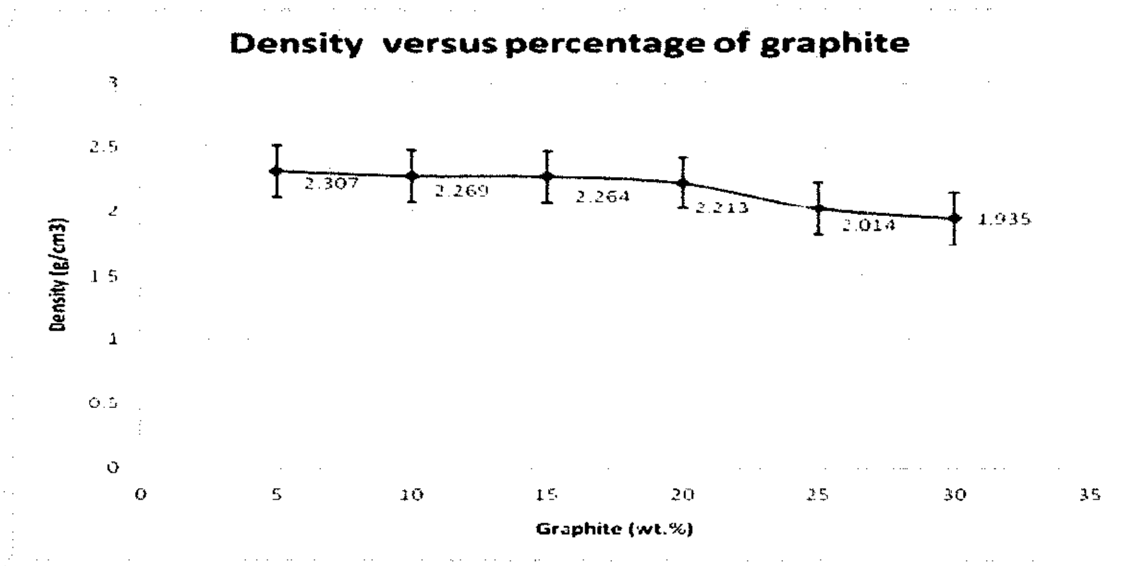


Figure 2.2: Graph of Density vs wt% of graphite in aluminium [3].

Graph shows a decrease in density as increasing amount of graphite weight percentage. Other than that, he also found that the hardness also increase with increasing amount of aluminium in AMCs.

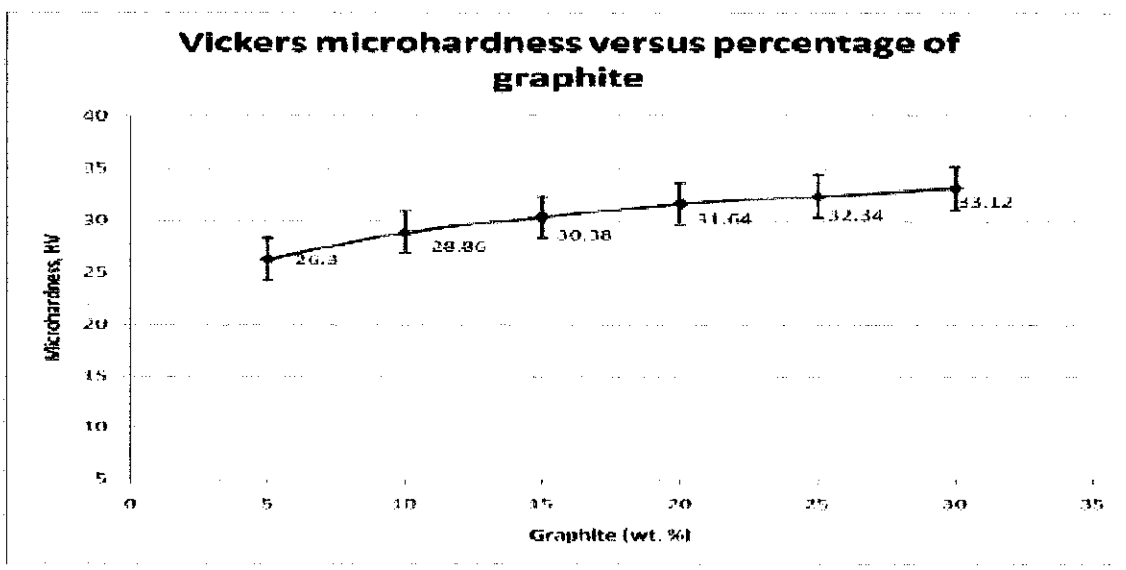


Figure 2.3: Graph of Vickers Microhardness vs wt% of graphite in aluminium [3].

Based on the objectives, the author also has done some research about wear behavior of a material. Wear has been defined as damage to a solid surface, generally involving progressive loss of material, due to relative motion between that surface and a contacting substance or substances. (ASTM, 1993) Experiment to observe wear behavior of AMCs reinforced with graphite has been conducted by experts before.

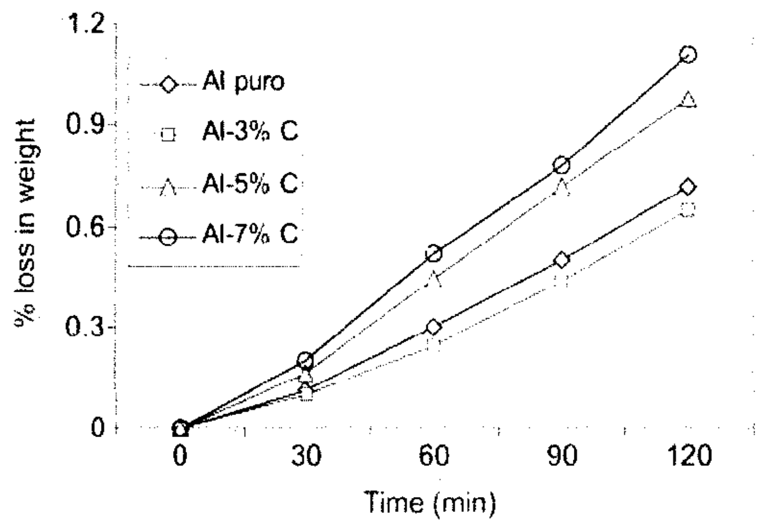


Figure 2.4: Graph of wear vs time for pure aluminium and aluminium-graphite [5].

From the graph, at 3% graphite content, the addition of graphite powder improves the tribological performance of the composite. However, when the graphite content is relatively high (above 3%), the wear endurance of the material decreases.

The other objective of this study is to observe the microstructure of the alloy powder particles within the samples of aluminium with different graphite content.

Further study based on experiment will be done by author to meet the objectives of the research. From the experiment that has been carried out by expert, these can be the reference to the ongoing project in term of determining the objective.

2.7 Powder Metallurgy (PM)

Metal matrix composite materials can be produced by many different techniques. The focus of the selection of suitable process engineering is the desired kind, quantity and distribution of the reinforcement components (particles and fibers), the matrix alloy and the application [5]. The powder metallurgy (PM) process is one of the most common methods for fabricating MMCs.

The advantage of PM method is the likelihood of fabricating high quality parts with complex shapes with high tolerance standards in a reasonably priced manner. PM offers compositional flexibility, least separatism effects and more likely to produce graded microstructures which differ in physical and mechanical properties. Also, PM offers economical advantages over ingot metallurgy in terms of cost, precision and productivity. PM process involves mixing, compacting and sintering.

Mixing refer to powders of different chemistries being combined. The absolute mixing of metal matrix powder and reinforcement ensures that the added reinforcement material are evenly distributed which facilities and the compaction process afterwards.

Compaction is the step where the mixed powders are pressed into various shapes in dies [7]. The purposes of compaction are to obtain the required shape, density, and particle-to-particle contact to make the part sufficiently strong for further processing [7]. The work part after pressing is called ***green compact***, the word green means that it is not yet fully processed. It fulfilled all the shape of the part required, but does not have sufficient strength as a working part since the powders are not bonded together.

Sintering is a process where green compacts are heated in a controlled-atmosphere furnace to a temperature below the melting point but sufficiently high to allow bonding of the individual particles [7]. After pressing, the green compact lacks strength and hardness. Sintering can be said as a heat treatment operation performed on the compact to bond its metallic particles, thereby increasing strength and hardness.

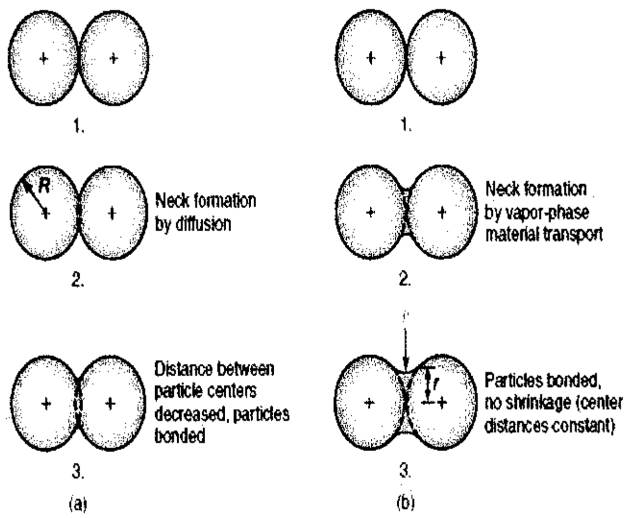


Figure 2.5: Sintering process [7].

The advantages and disadvantages of using powder metallurgy method of processing metal powder are:

Table 2.2: Advantages and disadvantages of powder metallurgy process.

Advantages	Disadvantages
PM parts can be mass produced to net shape or near net shape, complex or unique shapes of parts.	Metal powders do not readily flow laterally in the die during pressing and allowances must be provided for ejection of the part from the die after pressing.
Cost effective as it reduce machining process and material waste.	Difficulties with storing and handling metal powders (such as degradation of the metal over time, and fire hazards with particular metals).
Parts having a specified level of porosity, high purity and less foreign substances can be made.	Variations in material density throughout the part, especially for complex part geometries.
Part produce are of high strength and wear resistance.	High tooling equipment and material cost.

2.7.1 Hot Pressing

Hot pressing is a high-pressure, low-strain-rate powder metallurgy process for forming powder compact at a temperature high enough to induce sintering and creep processes [7]. This is achieved by the simultaneous application of heat and pressure. This method of powder metallurgy is usually used to fabricate hard and brittle material.

Hot press of metal powder is carried out in vacuum environment. In simpler way to explain, hot pressing is a powder compaction method involving uniaxial pressure applied to a controlled amount of powder placed in a die between two rigid rams. This process is carried out at elevated temperature and under vacuum, producing high purity parts with increased strength and high green density.

Inductive heating is the heating process that commonly used in the industry nowadays. It works by heating with high-frequency coils that can induce temperature rises. The mould is made out of graphite or steel, and pressure is applied by one or two cylinders onto the punches. The mould is placed in an induction coil. During sintering a high frequency generator and the induction coil generates heat in the mould.

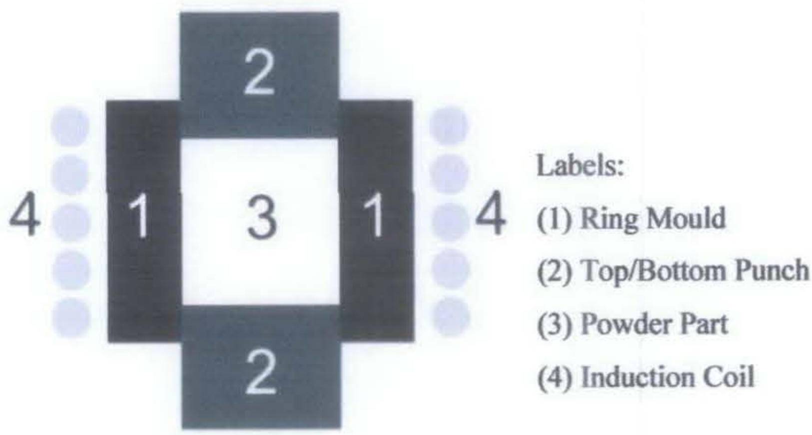


Figure 2.6: Inductive heating of hot pressing.

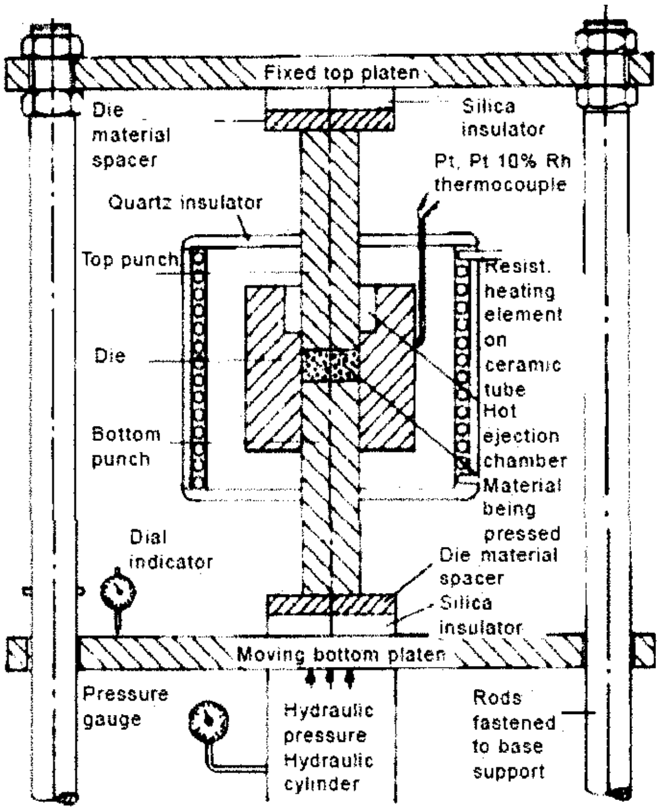


Figure 2.7: Schematic design of hot pressing unit [18].

The various steps involved in the hot pressing procedure are as follows:

1. Powder or a cold compacted preform is placed into the die mould.
2. The mould is heated either by resistance or by induction method to a predetermined temperature.
3. The powder in the die cavity is then pressurized.
4. The temperature is steadily increased during compacting until a maximum required temperature is reached;
5. Compacting pressure and temperature are maintained for a dwell time.
6. The mould is cooled slowly, under pressure, to a temperature at which oxidation of the material would not occur.

The advantages of hot pressing are it lowers the pressures required to reduce porosity and speeds welding and grain deformation processes [8]. Also it permits better dimensional control of the product, reduced sensitivity to physical characteristics of starting materials, and allows powder to be driven to higher densities than with cold pressing, resulting in higher strength [8].

2.8 Rule of Mixtures (ROM)

'Rules of Mixtures' are mathematical expressions which give some property of the composite in terms of the properties, quantity and arrangement of its constituents [17].

The properties of the composites, usually density can be predicted by the 'Rule of Mixtures' can be denoted as weight fraction or volume fractions. These expressions are derived for a two-phase material and then generalized to a multiphase material.

The volume fractions and weight fractions are given by the equation below.

$$v_c = v_r + v_m \qquad (2.5.1a)$$

$$Vr = \frac{v_r}{v_c}, \qquad V_m = \frac{v_m}{v_c} \qquad (2.5.1b)$$

and

$$w_c = w_r + w_m \qquad (2.5.1c)$$

$$W_r = \frac{w_r}{w_c}, \qquad W_m = \frac{w_m}{w_c} \qquad (2.5.1d)$$

The density of the composites material can be obtained in order to establish conversion relations between the weight fractions and the volume fractions. From the basic equation of density, the mass divided by the volume.

$$\rho = \frac{m}{v}$$

The weight in equation Eq. (2.5.1c) can be replaced by the density and volume and equation written as:

$$\rho_c v_c = \rho_r v_r + \rho_m v_m \qquad (2.5.2)$$

Dividing both sides in Eq. (2.5.2) by v_c and substituting Eq. (2.5.1b), the Eq. (2.5.2) can be rewritten as:

$$\rho_c = \rho_r V_r + \rho_m V_m \qquad (2.5.3)$$

For the case of fiber-matrix composites, the equation Eq. (2.5.3) can be written as:

$$\begin{aligned} \rho_c &= \rho_r V_r + \rho_m V_m \\ &= \rho_r V_r + \rho_m (1 - V_r) \\ &= V_r(\rho_r - \rho_m) + \rho_m \end{aligned} \qquad (2.5.4)$$

Since $V_r + V_m = 1$

where;

v_c, v_r, v_m	represent the volume of composite, fiber and matrix material.
V_r And V_m	represent volume fraction of fiber and matrix material.
w_c, w_r, w_m	represent weight of composite, fiber and matrix material.
W_r, W_m	represent weight fraction of fiber and matrix material.

CHAPTER 3
METHODOLOGY

The project work can be divided into two parts which is for the first semester and work for the second semester. In the first semester, the project work focuses more on literature reviews. Besides, material preparation for experiment also becomes one of the major tasks in this semester. After some researches, the author makes the research flowchart to get more clearly about the sequence for this project.

The project begins with gathering all information by researching and understanding on the fundamentals of metal matrix composites, powder metallurgy process and sample preparation for testing. All information about the materials, equipment and experimental procedure were gathered in the first semester of the final year project.

Next step of this project is the preparation of samples by powder metallurgy technique. As of the research flowchart, the investigation into the density, microstructure, hardness, friction, and wear mechanisms of the aluminium metal matrix reinforced with graphite will begin with researches. The author had read journals and articles to enhance the understanding on materials, microstructure, friction, density, wear mechanisms and all equipments that can be used on her project.

All the experimental work is done in Block 17 of Mechanical Engineering Building with the assistance of mechanical technicians.

The project flow of this research is summarized in Figure 3.1 below.

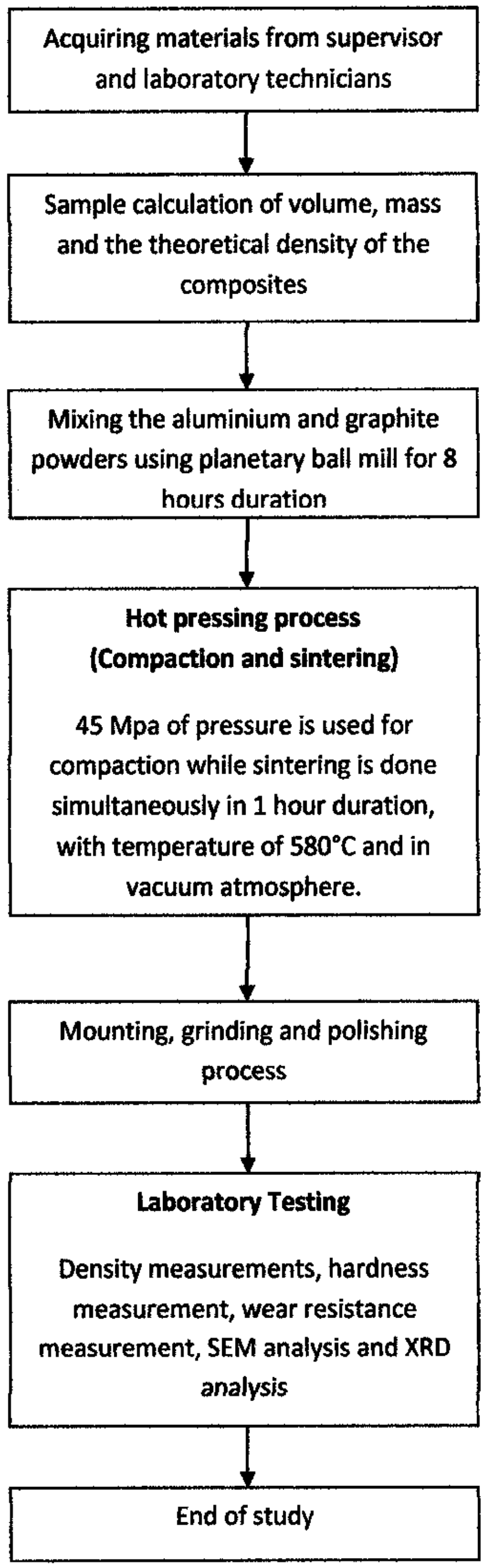


Figure 3.1: Project flow chart.

3.1 Samples Preparation

Figure 3.2: Aluminium powder.

Figure 3.3: Graphite powder.

The experiment starts with calculating the amount of powder needed for the composition of 2wt%, 4wt%, 6wt% and 8wt%. The diameter and height of a pallet is 40mm and 5mm respectively. The density of aluminum is 2.70 g/cm^3 and graphite is 2.26 g/cm^3. The mass and theoretical density of the composites samples were calculated using Rule of Mixture (ROM). The example of calculation is shown in APPENDIX B. The result from all the computation can be referred at table below.

Table 3.1: Mass of powder required for sample preparation.

No	Graphite wt%	Mass of Aluminium (g)	Mass of Graphite (g)
1	0	16.946	0
2	2	16.624	0.284
3	4	16.378	0.568
4	6	16.094	0.852
5	8	15.810	1.136
TOTAL		81.852	2.840

After calculation is done, the process of powder metallurgy begins with the mixing of aluminium powder with graphite particle at different weight fraction of graphite; 2wt%, 4wt%, 6wt%, and 8wt%. The mixing process is done using Ball Mill. Aluminium and Graphite powder will go through mixing process with 8 hours [3] duration to make sure that the mixture is homogenized.

Figure 3.4: Ball mill.

After that, the compaction and sintering process is done simultaneously using Hot Press machine which follow standard of ASTM B331-95 for Standard Test Method for Compressibility of Metal Powders in Uniaxial Compaction. Pressure of 6500lb (45MPa) is applied to the powders to form them into required shape. The author use 45MPa compaction forces because based on the mechanical properties of aluminium, it only can sustain load within 40-50 MPa.

As for the sintering temperature, sintering is usually evident at temperatures in excess of approximately two-third of the absolute melting temperature of aluminium [9]. Since the melting temperature of aluminium is 660°C, the author decided to use 580°C as the sintering temperature for the sample. The compaction and sintering is done within 60 minutes duration. As stated in literature review, the process is carried out in vacuum environment.

The mixture of aluminium and graphite powder will be placed inside the mould, which is between the upper and lower puncher. The door of furnace will be closed and the process of producing compacted samples will be started.

Figure 3.5: Hot press machine.

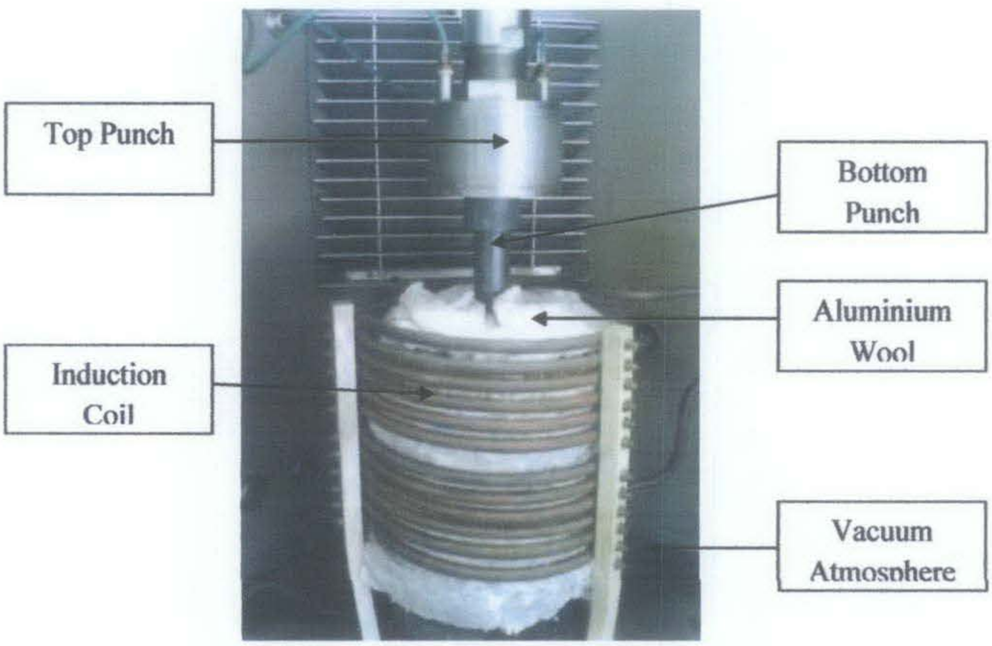


Figure 3.6: Part of hot pressing for inductive heating.

3.1.1 Hot Mounting

After the samples were sintered, the samples were then mounted by using Buehler, Simpliment 1000 mounting machine. Thermosetting polymeric powder used is phenolic powder. The phenolic powder is placed in the mould with samples then was heated for 2min and cooled for 5min under the pressure of 3500psi.

Figure 3.7: Auto mounting pressing machine.

3.1.2 Grinding and Polishing

Grinding and polishing process were performed by using Grinder and Polisher machine model Metaserv 2000. The samples were ground with SiC paper and running water. The SiC paper used raging from 320 grits to 2400 grits. The samples are polished with the rough polish started with 6micron and then with the 1 micron diamond compound. The speed for both processes is 150 to 200 rpm.

Figure 3.8: Grinder and polisher machine.

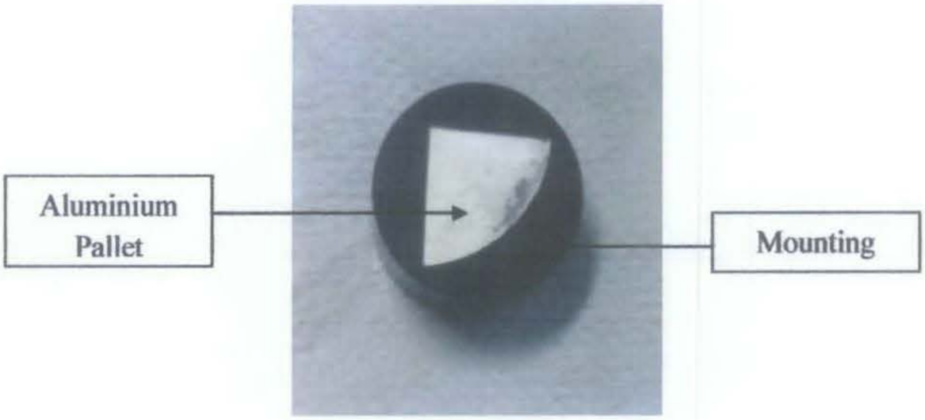


Figure 3.9: Finish sample.

3.2 Sample Testing

3.2.1 Density Measurement

The density of a material is defined as its mass per unit volume. Density of each sample which contains different percentage of graphite amount in aluminium will be measured using density measurement machine namely Mettler Toledo AX205 and the measurement perform using Archimedes Principles. It is expected that the density will decrease as the amount of graphite's weight fraction in aluminium metal matrix composites increase.

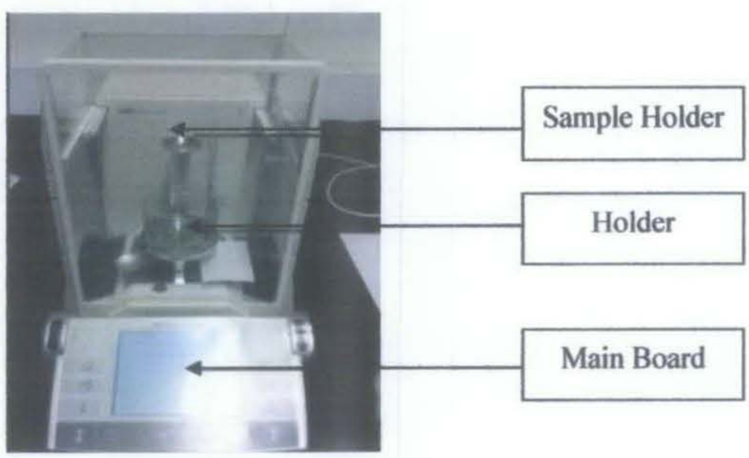


Figure 3.10: Density measurement device.

3.2.2 Microstructural Analysis

Microstructures, physical and mechanical properties of each samples will be analyze using Scanning Electron Microscope. The **scanning electron microscope (SEM)** is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern [12]. SEM has a magnification range from 15x to 200,000x and a resolution of 5 nanometers. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time as well as the actual strikingly clear images. The testing using SEM will follow E 986 – 04 Standard Practice for Scanning Electron Microscope Beam Size Characterization. Result will be compared and it is expected that all the characterization improved with the increasing of graphite amount.

Figure 3.11: Scanning Electron Microscope (SEM).

Other than that, the author also done X-Ray Powder Diffraction Analysis of aluminium based graphite particles composite. **X-ray powder diffraction** (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined. It is most widely used for the recognition of unknown crystalline materials (e.g. minerals, inorganic compounds).

3.2.3 Hardness Measurement

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting. The Vickers test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter, and the indenter can be used for all materials irrespective of hardness [13].

Hardness of each samples contains different percentage of graphite amount will be measured using Vickers Microhardness Test Machine. The testing follows ASTM E92-82 Standard Test Method for Vickers Hardness of Metallic Materials. Since AMCs is combined with the high tensile strength and modulus of elasticity of graphite, thus achieving good strength-to-weight and modulus-to-weight ratios in the resulting composite material. Therefore, it is expected that the hardness of each sample increase as the amount of graphite increase.

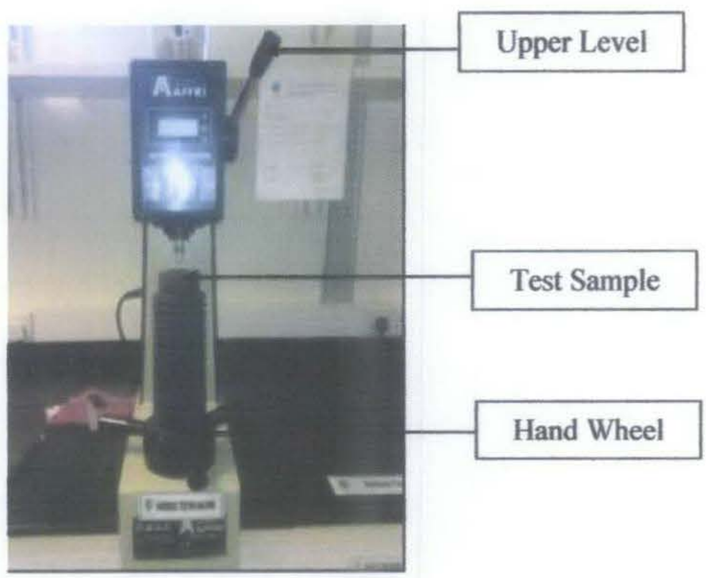


Figure 3.12: Vickers microhardness test machine.

3.2.4 Wear Resistance Observation

In this research, it is expected that graphite in aluminium alloys would improve friction and wear performances. The wear test will be carried out in a disc-shaped device. The pin on-disc machine is a custom built apparatus used to test materials in sliding contact over a range of load and speed. The pin consists of a piece of cylindrical aluminium. The disc material may typically be steel. In the pin-on disc tribometry, a pin is loaded onto the test sample (disc) with a precisely known weight and the disc is rotated. Wear coefficients for the pin and disc materials are calculated from the volume or weight of the material lost during the test.

For wear testing, the DUCOM Multi Specimen Tester is to characterize sliding contact between two samples over a wide range of test parameters. The wear behaviour result will be obtained in the form of coefficient of friction, weight loss and wear rate.

The samples will be subjected to wear cycles with 48 HRC stainless steel disc at 120 rpm and a load of 60N without lubrication, for 3 minutes. The author will measure the loss in weight, as Archard et. al. (1953) proposed in his model of wearing.

Figure 3.13: DUCOM multi specimen tester.

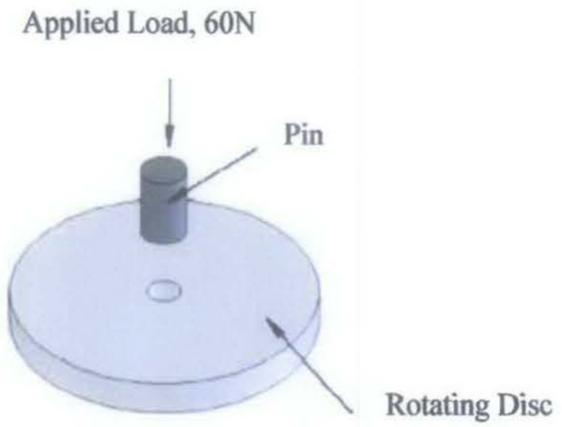


Figure 3.14: Schematic presentation of pin-on-disc wear experiment [17].

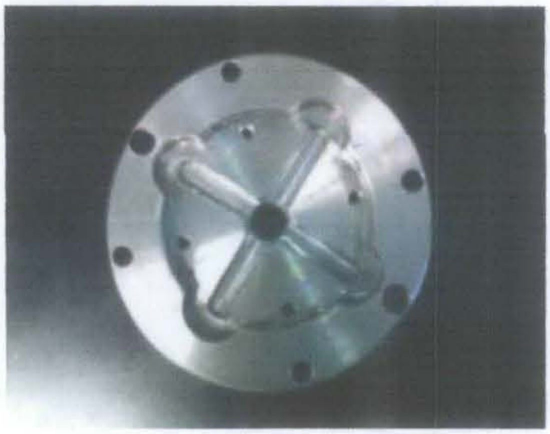


Figure 3.15: Disc holder.

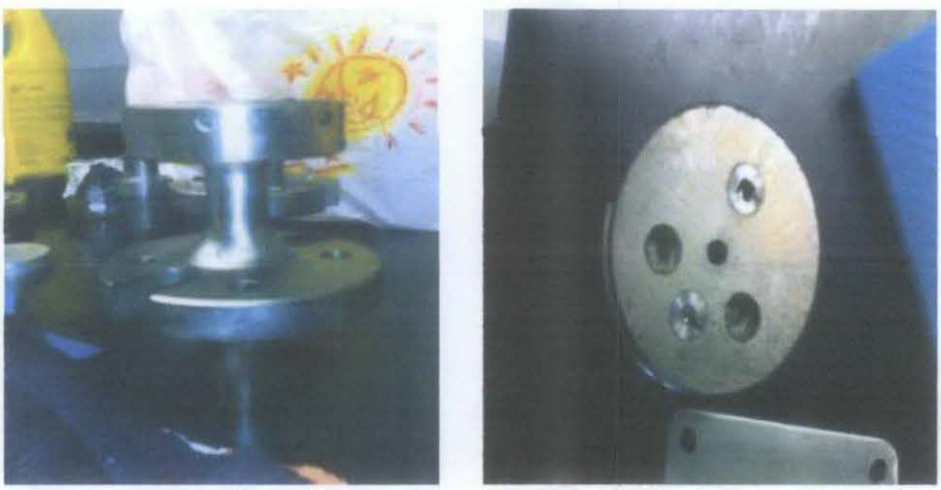


Figure 3.16: Side and front view of pin holder.

Figure 3.17: Inside view of the DUCOM Multi Specimen Tester.

To prepare the pin, the author has to cut the samples using abrasive cutter into 12mm X 5mm X 5mm in dimension.

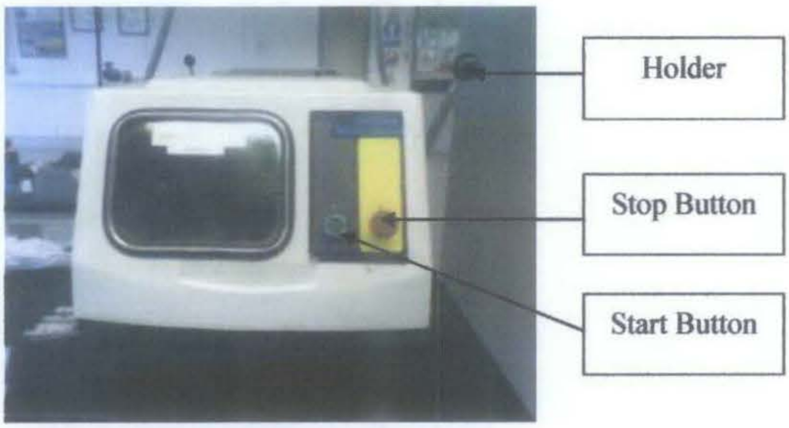


Figure 3.18: Abrasive cutter.

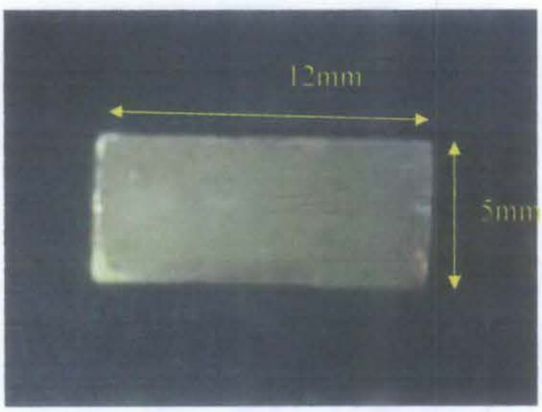


Figure 3.19: Sample of pin.

3.6 Data Analysis

After all the testing done, the analysis of the result will be conducted. The most important task to be analyzed to achieve the objective for this project are measuring the compacted density, hardness, wear resistance and observing its microstructure improvement of the samples.

Table 3.2: Project Milestone for Final Year Project II.

NO	ACTIVITIES	DETAIL	WEEK 1	2	3	4	5	6	7	MID SEMESTER BREAK	8	9	10	11	12	13	14	15
1	Literature review	Study on testing required on samples	■	■	■													
2	Literature review	Study on microstructure and elemental analysis	■	■	■	☆												
3	Project work	Sample set up		■	■	■												
4	Project work	Density and Hardness testing				■	■	■	■									
5	FYP documentation	Submission of progress report									☆							
6	Project work	XRD analysis										■						
7	Project work	FESEM and EDEX analysis										■						
8	Project work	Fabricating pallet holder for wear testing									■	■						
9	Project work	Wear resistance testing										■						
10	FYP documentation	Data gathering and analysis										■	■					
11	FYP documentation	Pre-EDX												☆				
12	FYP documentation	Submission of draft report													☆			
13	FYP documentation	Submission of project dissertation (soft bound)														☆		
14	FYP documentation	Submission of technical paper														☆		
15	FYP documentation	Oral Presentation															■	■
16	FYP documentation	Submission of project dissertation (hard bound)																☆

☆ - Suggested milestone ■ - Process

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Diameter, Thickness and Density Measurement

After performing hot press process, the diameter, thickness and density of each samples was measured. Diameter and thickness were measured using vernier caliper while density is measured using Mettler Toledo machine. The data recorded is shown in APPENDIX C. Plot of density for Al-Graphite for various weight percentage of graphite reinforcement is presented in Figure 4.1 below.

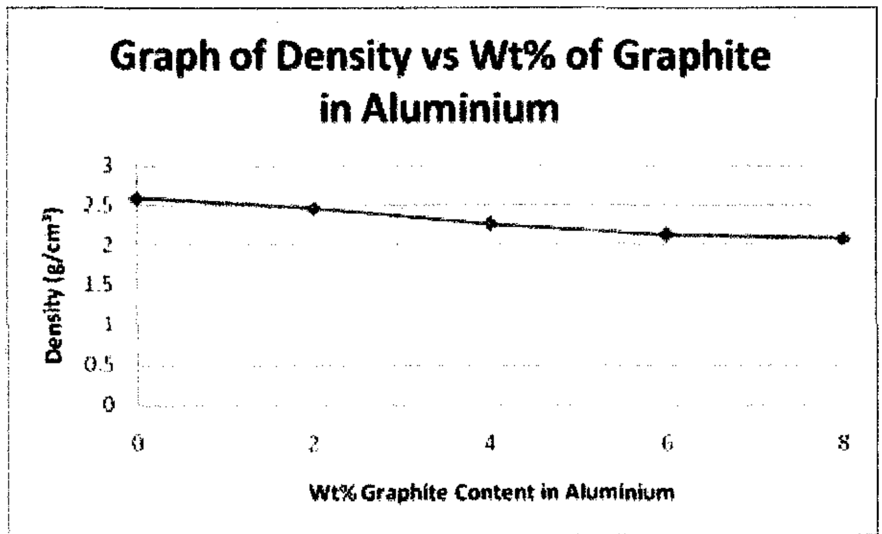


Figure 4.1: Graph of density vs wt% of graphite in aluminium.

From the graph, it can be seen that by adding graphite particulate in aluminium matrix composite may caused the density of the composites to decrease. The microstructure of graphite reinforced in the aluminium matrix provides more empty spaces between molecules of the fabricated samples. This means that empty spaces increase as the weight percentage of graphite in aluminium matrix increase which later decreases the density of aluminium MMC produced.

Other than that, since the density of graphite itself is lower than that aluminium, it can be said as the amount of weight percentage of graphite in the composite increase, the weight percentage of aluminium itself decreased. Thus, the density of the fabricated composite decreased.

According to Fuentes et al. (2003), the density of Al-Graphite with 7% carbon is about 1.86 g/cm^3. Thus, referring to the result obtained by the author, it can be said that the densities of the composites sample with various amount of graphite is dominated by pure aluminium itself [5].

The finding is actually a good news for automotive and aircraft industry. This is because, as the amount of aluminium matrix's can be reduced by replacing it with graphite, the cost of raw material that used to fabricate the body of automotive and aircraft can be reduced. Substantial saving is predicted in fabricating the body since aluminium price currently is $9215 USD per ton and graphite is only $2500 USD per ton. Thus, by decreasing the amount of aluminium used yet the density decrease by adding graphite, substantial savings can be done.

4.2 Hardness Measurement

The hardness testing was done for each aluminium samples with different weight percentage of graphite using Vickers Microhardness Testing Machine. The data is tabulated and is shown in APPENDIX C. Plot of Vickers Hardness for Al-Graphite for various weight percentage of graphite reinforcement is presented in Figure 4.2 below.

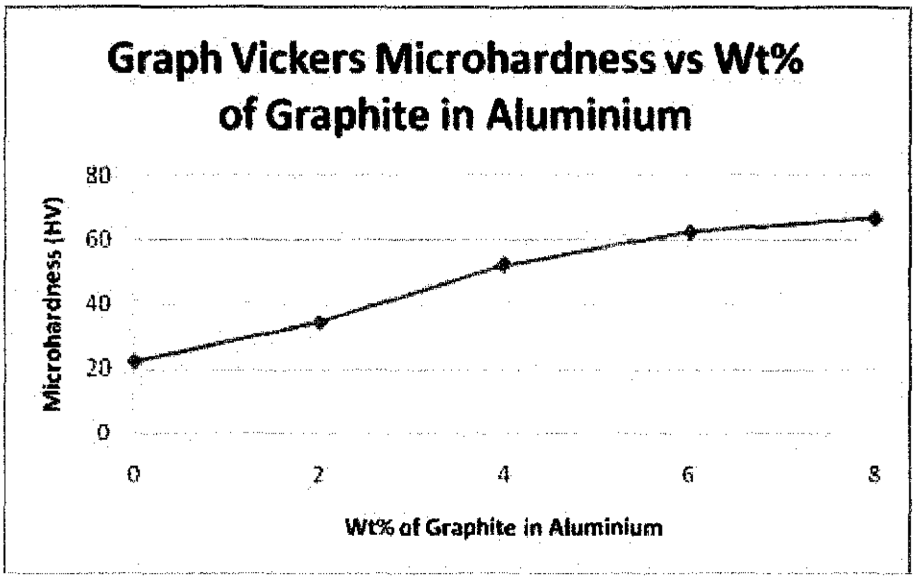


Figure 4.2: Graph of Vickers Microhardness vs wt% of graphite in aluminium.

From the graph, it can be seen that by adding graphite particulate in aluminium matrix composite may caused the hardness of the composites increased. A maximum hardness of 66.7 HV occurs at 8% weight of graphite particle in aluminium matrix whereas unreinforced aluminium has the lowest amount of hardness which is 22.2 HV.

According to Chen et al. (2008), clean reinforcement matrix interfaces improve interfacial bonding strength and homogeneous dispersed reinforcing particulate result in better hardness of aluminium matrix composites [15].

Other than that, according to Rodr'ıguez et. al. (2006), microstructure and mechanical response of the matrices is modified due to the reinforcement. The grain size of the matrices is reduced respect to the unreinforced alloys, it is observed a higher dislocation density in the matrices and the nucleation of incoherent precipitates is favored by the presence of reinforcements due to the higher defect's density. For these reasons the matrices are expected to be harder than the unreinforced alloys [17].

Eventhough hot pressing was used to compact and sinter the composite at the same time where it may decrease the porosities compared to conventional technique, the porosities are still present. As we know, porosity that contained in compacted composites may cause it has low in hardness. Since the graphite particles (64μm) are smaller than the size of aluminium particles (75μm), the graphite particle is said to fill the porosity that occurs within the aluminium matrix. So, as the amount graphite particles in aluminium matrix increase, more porosity would be filled which later may improve the hardness of the composites.

4.3 Wear Resistance Observation

The wear resistance testing was done for each aluminium samples with different weight percentage of graphite using DUCOM Multi specimen Tester. The data is tabulated and shown in APPENDIX C. Plot of % of weight loss vs time for Al-Graphite for various weight percentage of graphite reinforcement is presented in Figure 4.3 below.

Figure 4.3: Graph of % loss in weight vs time for Al-Graphite for various weight percentage of graphite reinforcement.

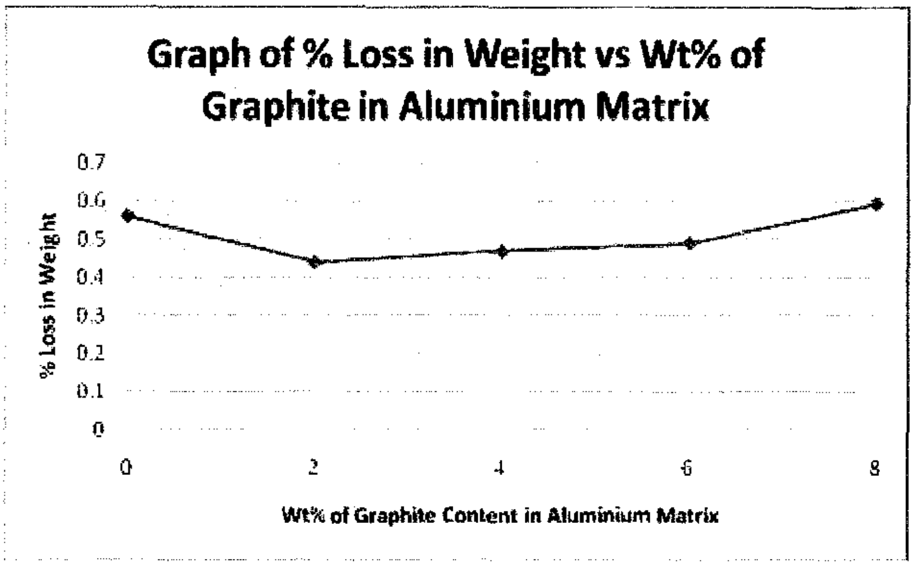


Figure 4.4: Graph of % loss in weight vs wt% of graphite content in aluminium matrix.

From the graph, at 2% graphite content, the addition of graphite powder improves the tribological performance of the composite. However, as the graphite content is increased (above 2%), the wear endurance of the material decreases.

This clearly indicates that the composites behave as two phases system: when a little graphite powder is added to the Al-based matrix, and in spite of the poor interfacial interaction, the wear resistance of the matrix itself is slightly improved by the presence of the few particles of graphite scattered throughout the matrix.

As the amount of graphite is increased, the wear is endured basically by the graphite alone, with little participation of the Al alloy matrix, and since no interaction exist between the two phases, the composites behave almost as a granular structure, with little wear resistance.

Although this wear behavior results obtained by the author did not achieve the research's objective, but for the author it is a good finding. From the result, we may estimate the optimum amount of graphite to be added in the aluminium matrix in order to improve wear resistance of pure aluminium which is below 6%. For best result, we only need to add 2% of graphite in aluminium matrix.

4.4 Scanning Electron Microscope (SEM) Analysis

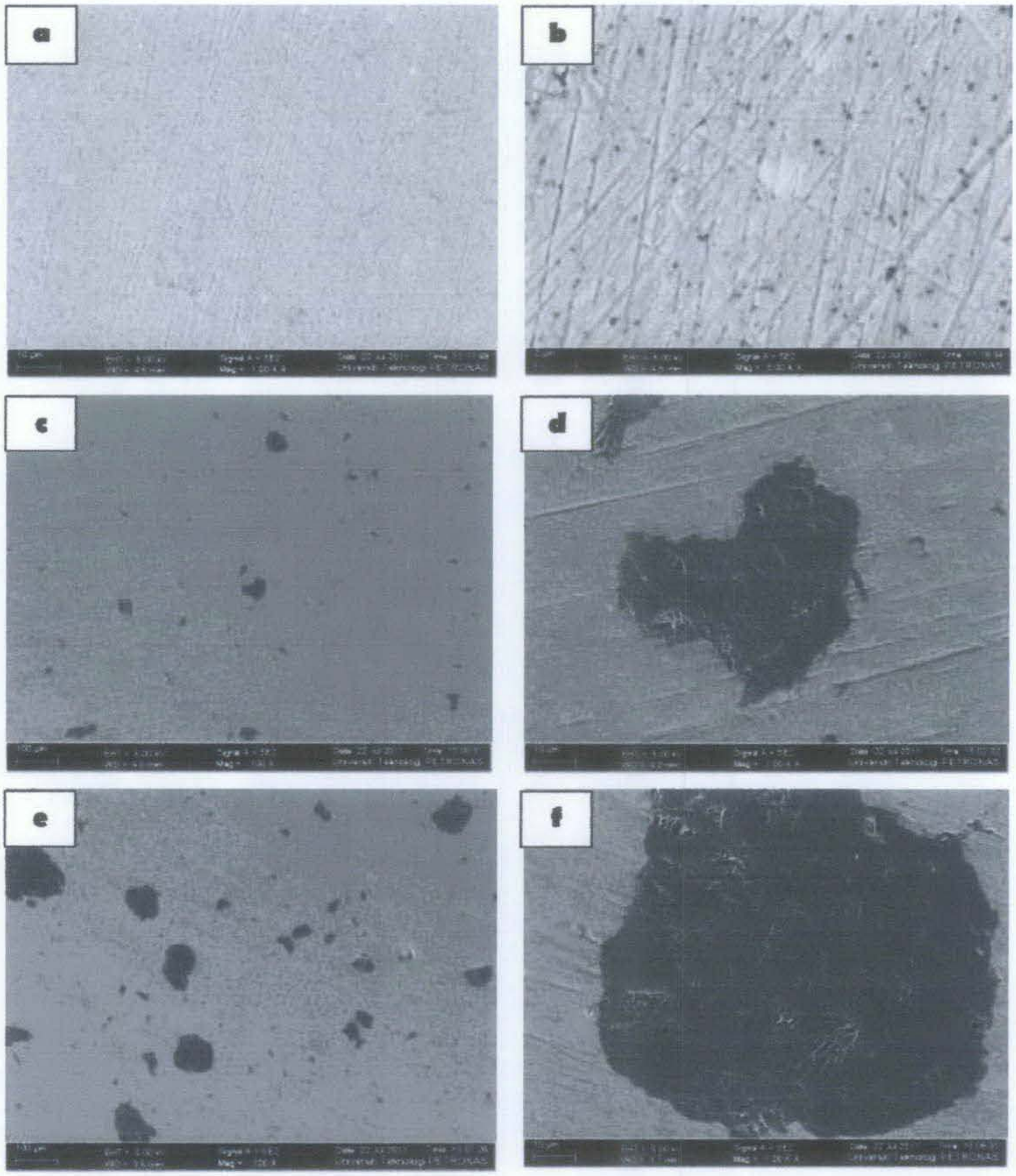


Figure 4.5: Showing SEM images for a) Pure Al 1000x, b) Pure Al 5000x, c) Al-2wt% Graphite 100x, d) Al-2wt% Graphite 1000x, e) Al-4wt% Graphite 100x, f) Al- 4wt% Graphite 1000x.

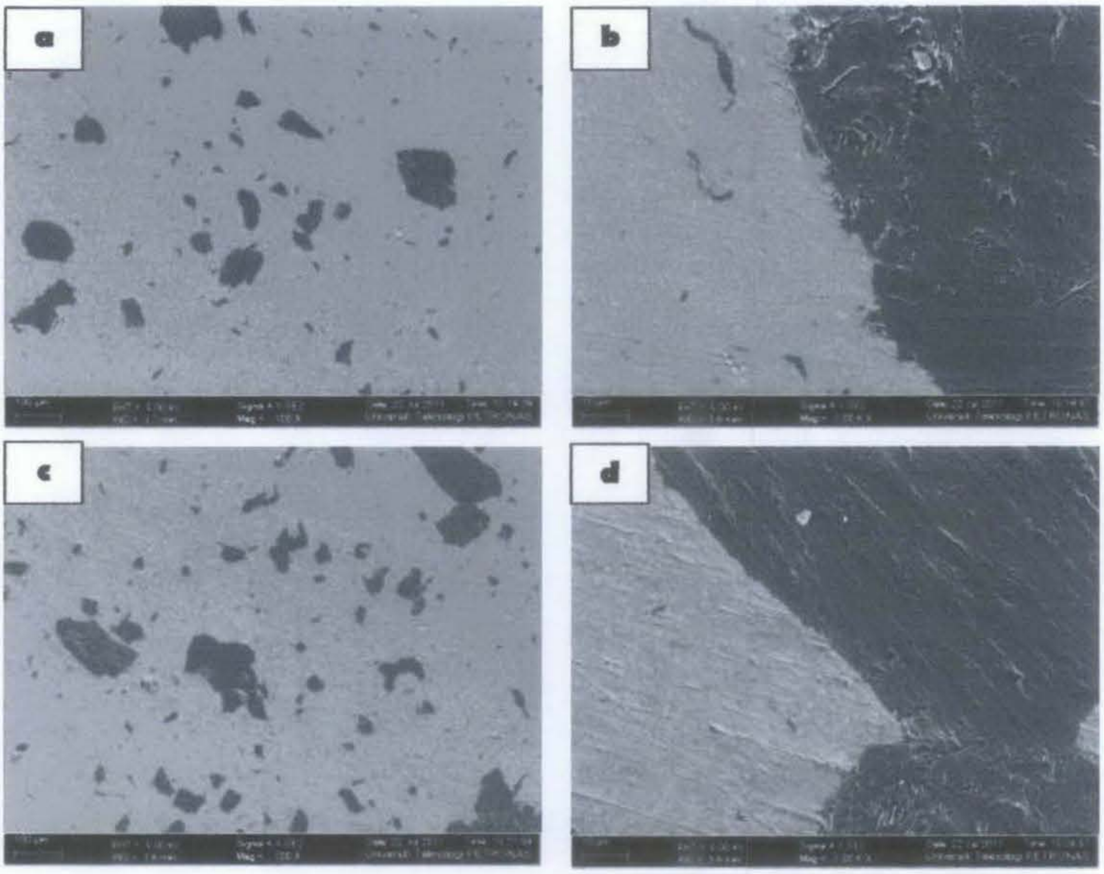


Figure 4.6: Showing SEM images for a) Al- 6wt% Graphite 100x, b) Al- 6wt% Graphite 1000x, c) Al- 8wt% Graphite 100x, d) Al- 8wt% Graphite 1000x.

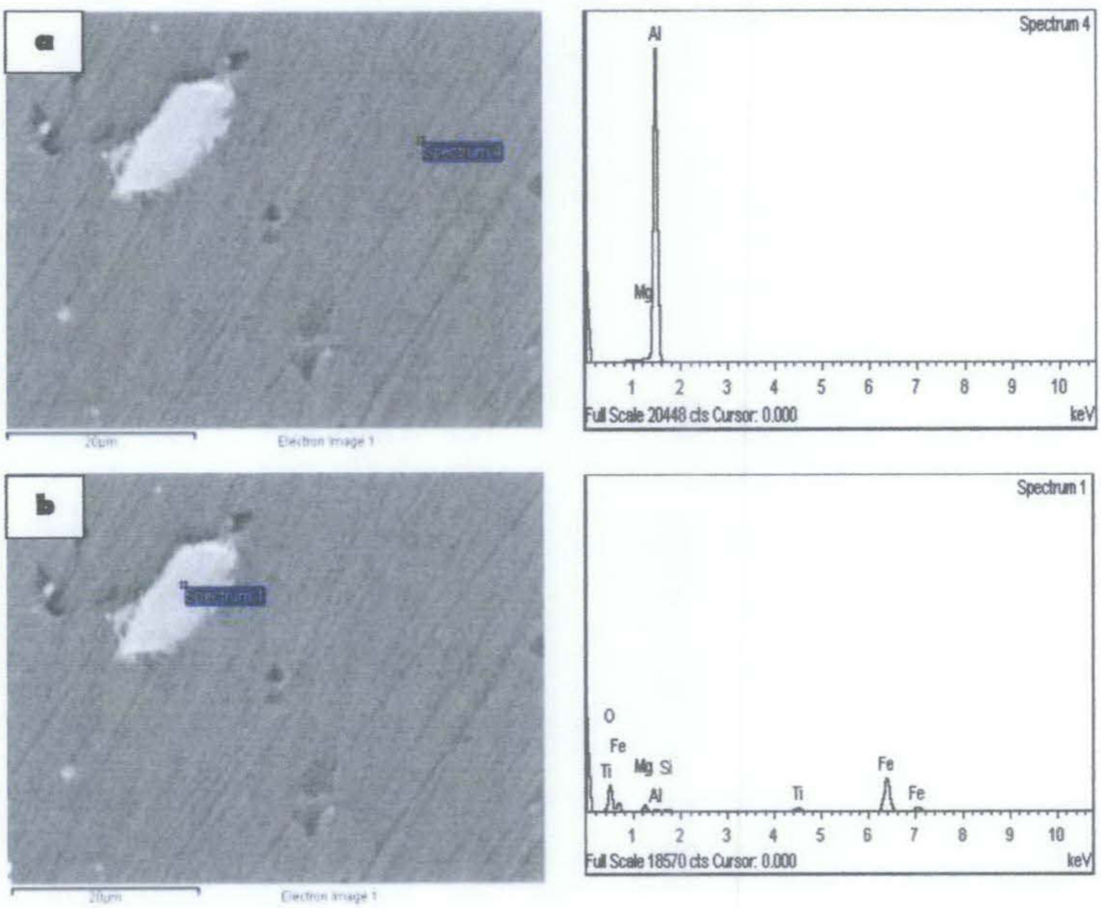
The light color in the images represent aluminium phase and the dark color represent graphite. From the image we can see that dark color (graphite) distribution inside the images increase as the amount of graphite in the aluminium matrix increase. It represents the graphite distribution among the aluminium matrix. It is clearly that the graphite distribution can be said as uniform and homogenously distributed. It can be stated that the blending process via planetary ball mill which employed in this research was very effective for the dispersion of graphite particulate.

From all the images in Figure 4.5 and Figure 4.6 above, it can be said that porosity almost cannot be seen. That suits the advantage of fabricating the composites using hot pressing method which less porosity will occurred. But still, there is porosity contained in the composites, perhaps higher magnification are needed in order to see the porosity.

From the entire image for the magnification of 1000x shown above, it can be seen that graphite particulates are properly bonded with aluminium matrix and well continuance between each other. This condition formed good initial strength to the matrix, as confirmed by the results of microhardness testing. As the result, this can be the reason behind the strength that has been enhanced.

4.5 Energy Dispersive X-ray Spectroscopy (EDXS) Analysis

EDS analysis was done where the different points have been taken to verify the description of FESEM analysis above.



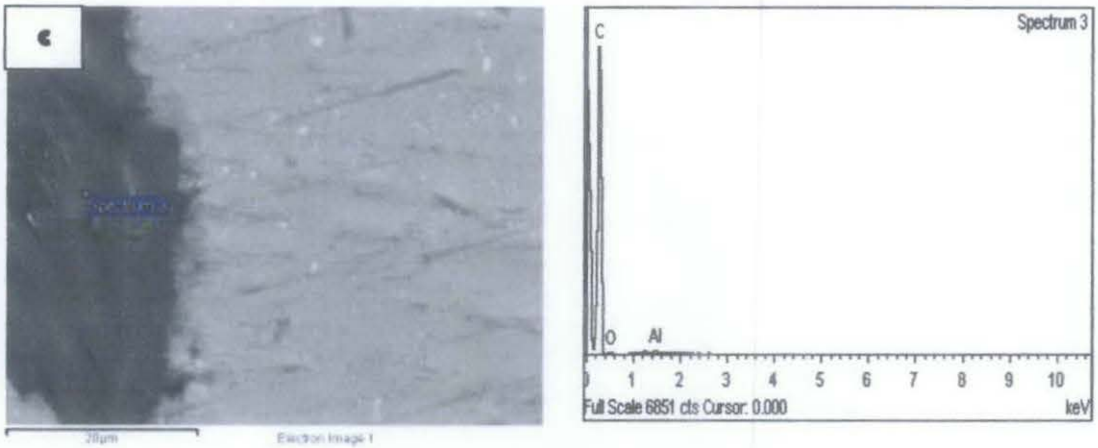
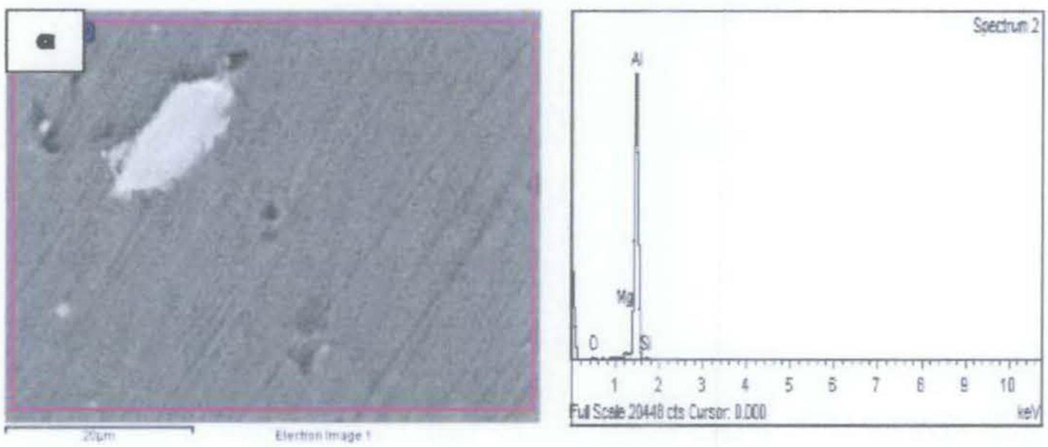
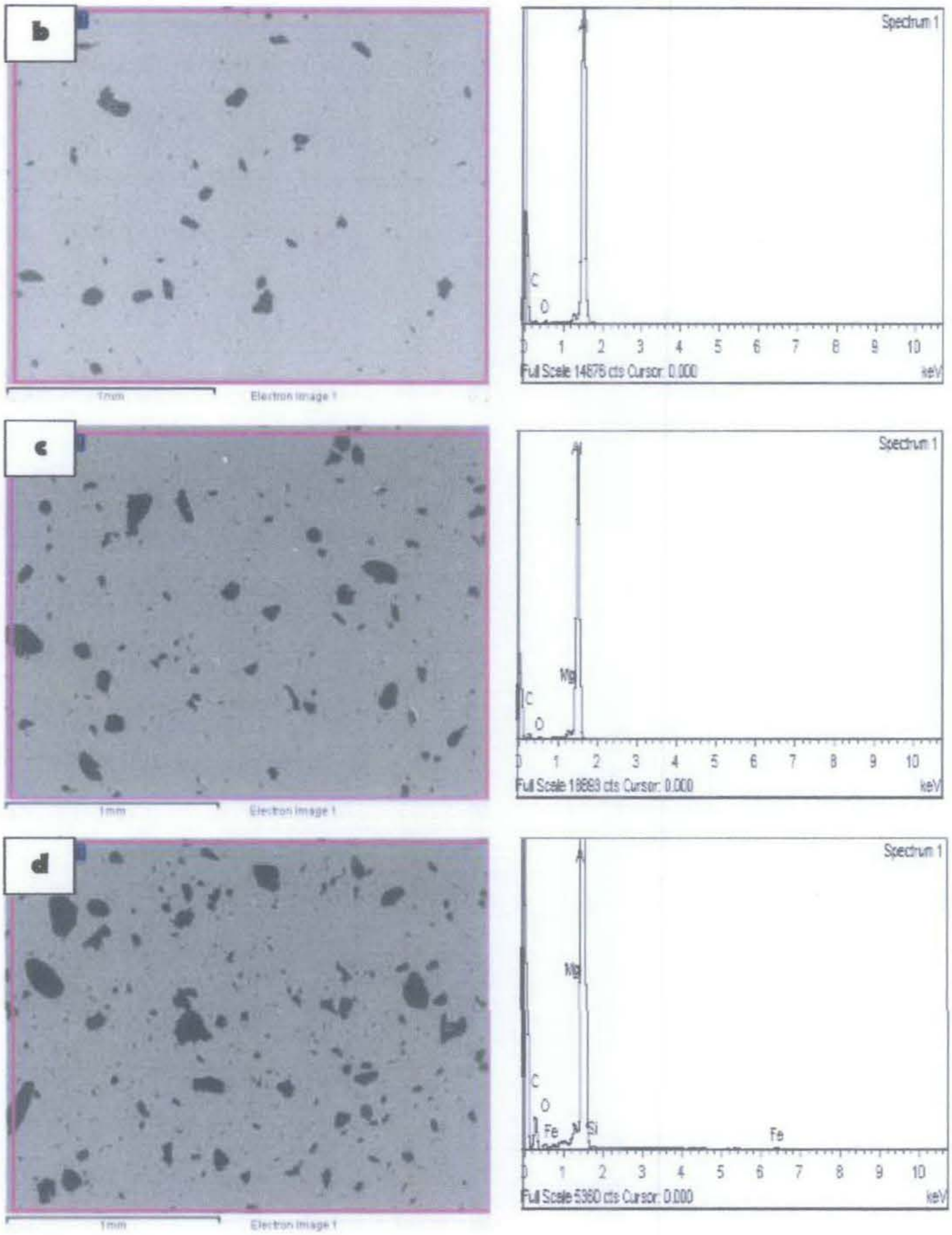


Figure 4.7: Showing qualitative result for a) Spectrum 4, b) Spectrum 1, c) Spectrum 3.

The grayish spectrum in Figure 4.7(a) represents aluminium. The lightest spectrum in Figure 4.7(b) represents the mixture of titanium, magnesium, iron, and silica that usually added in aluminium alloy to further strengthen the aluminium. All the fabricated composites contain added materials as mentioned before. As for the darkest spectrum in Figure 4.7(c), it represents graphite.

4.5.1 Analysis for 2wt%, 4wt%, 6wt% and 8wt% of Graphite in Aluminium Matrix





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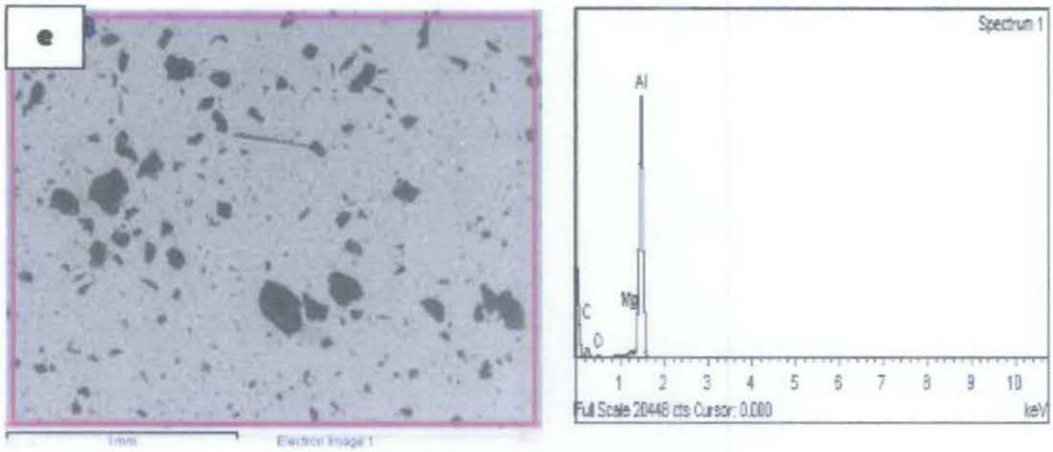

Figure 4.8: Showing qualitative EDS result for a) Al-2% Graphite, b) Al-4% Graphite, c) Al-6% Graphite, d)Al- 8% Graphite.

Figure 4.8(a), shows pure aluminium contains 78% Al for that spot only. Figure 4.8(b) shows aluminium with 2wt% of graphite contains 68% of aluminium and 26% of graphite. Figure 4.8(c) shows aluminium with 4wt% of graphite contains 61% of aluminium and 35% of graphite. Figure 4.8(d) shows aluminium with 6wt% of graphite contains 51% of aluminium and 45% of graphite. And lastly, Figure 4.8(e) shows aluminium with 8wt% of graphite contains 48% of aluminium and 50% of graphite. Based on qualitative result from Figure 4.8 above, it has been proven that graphite weight percentage in aluminium matrix composites is increased and the weight percentage of aluminium decreased.

It has been proven that the lightest spectrum in the image represents mixture of titanium, magnesium, iron, and silica that usually added in aluminium alloy to further strengthen the aluminium. All the fabricated composites contain added materials as mentioned above. Other than that, the existence of aluminium (grayish spectrum) and graphite (darkest spectrum) also has been proven.

4.6 X-Ray Diffraction Analysis (XRD)

XRD characterizations of Al-graphite composites with various percentage of graphite content were conducted in order to further understand the structure of the composites.

From Figure 4.9 below, it can be seen that as percentage of graphite in aluminium increase, the area under the aluminium curve decrease and area under graphite peak's curve increase. Area under the peak's curve represents the intensity of the materials contained in the composites. This means that it has been proven that amount of aluminium decrease and the amount of graphite increase.

It has also been observed from the XRD results that there is no structural changes occur during fabrication process and graphite particles have the same crystalline structure and are homogeneously distributed in Aluminium matrix which increases the mechanical properties of the composites.

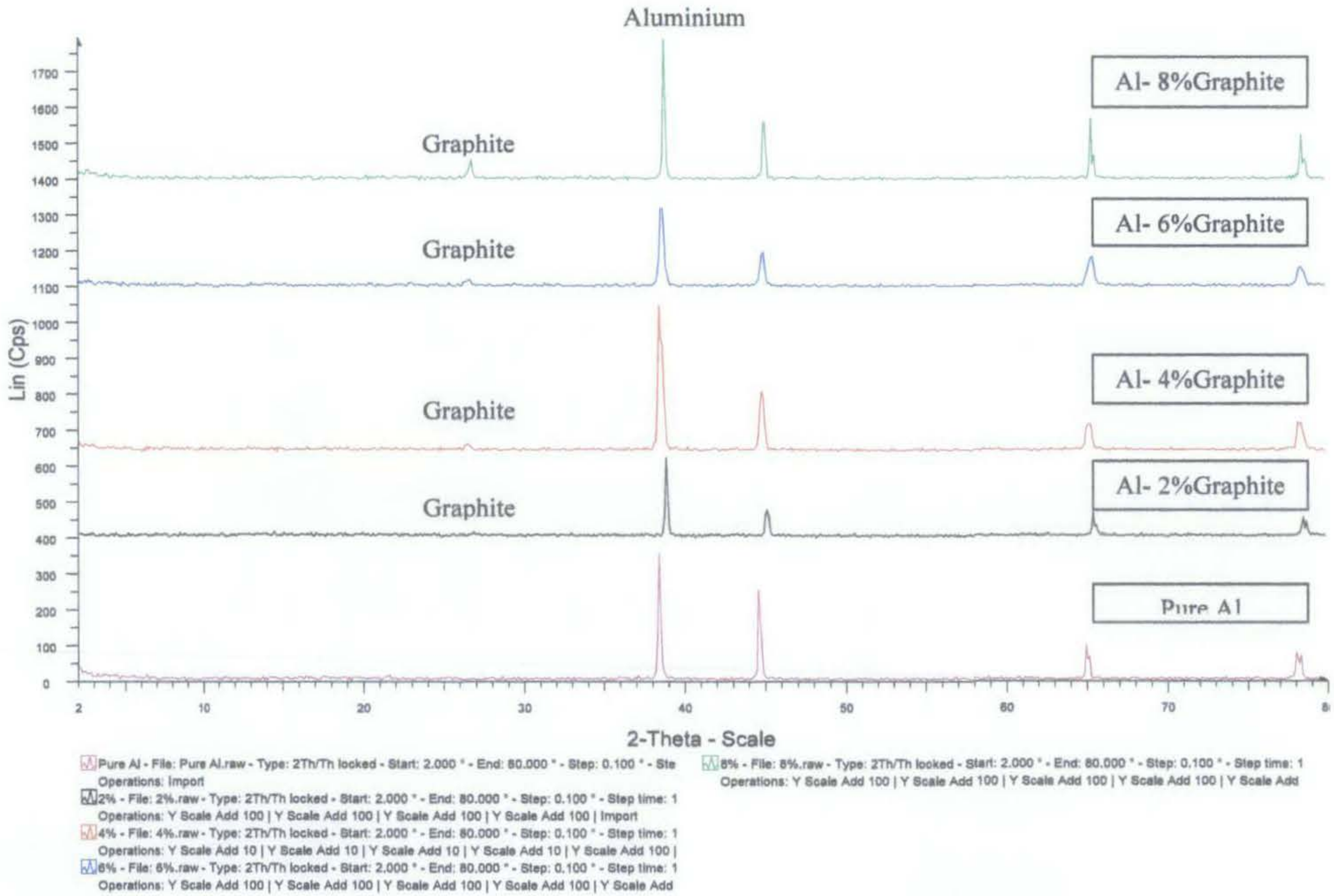

Figure 4.9: XRD patterns of different percentage of graphite reinforced aluminium matrix composites.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From the experiment done by the author, it is proven that the density of samples decrease as the weight percentage of graphite in aluminium increase. Other than that, the hardness also increases as the weight percentage of graphite in aluminium increase.

As for the wear resistance testing, the results obtained might not as expected by the author. The wear resistance of aluminium was not increase as the amount of graphite in the composites increase. But from the result, we may estimate the optimum amount of graphite to be added in the aluminium matrix in order to improve wear resistance of pure aluminium which is below 6%. For best result, we only need to add 2% of graphite in aluminium matrix.

From FESEM analysis, it is clearly seen that the graphite distribution can be said as uniform and homogenously distributed. Besides, from the entire image for the magnification of 1000x shown, it can be seen that graphite particulates are properly bonded with aluminium matrix and well continuance between each other. This condition formed good initial strength to the matrix, as confirmed by the results of microhardness testing. As a conclusion, it can be said that the microstructure behaviors reflect the hardness of the composite.

As for EDS analysis, it has been proven that the lightest spectrum in the image represents mixture of titanium, magnesium, iron, and silica that usually added in aluminium alloy to further strengthen the aluminium. Other than that, the existence of aluminium (grayish spectrum) and graphite (darkest spectrum) also has been proven. Besides, EDS analysis also shows the proved that graphite weight percentage in aluminium matrix composites are increased and the weight percentage of aluminium decreased.

5.2 Recommendation

For further research for the future, the survey needs to be in a larger scope. It is recommended for future investigation to:

1. Use the aluminium matrix reinforced with graphite which may cause substantial savings of raw materials cost for the vehicle's body manufacturing.
    - Since the first and second objectives has been proven right, it is recommended by the author to the automotive and aircraft industry to use aluminium matrix reinforced with graphite to save cost.

2. To fabricate aluminium metal matrix composites reinforced with graphite using hot pressing method.
    - The author has seen some of her friends that used cold compression method to fabricate the composites. As the result, the finish samples contain lots of porosity which makes it brittle and low in strength.
    - Samples that fabricated using hot pressing, it contains low porosity, which later makes the samples high in strength have good wear resistance.

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APPENDIX A

PHYSICAL PROPERTIES OF RAW METAL POWDERS

1.0 Aluminium (Al)

Physical properties of aluminium.

Properties	Aluminium
Density (g/cm^3)	2.7
Young's Modulus (GPa)	70
Hardness (MPa)	420
Tensile Strength (MPa)	230 – 570
Compression Strength (MPa)	530
Thermal Conductivity (W/m.K)	237

2.0 Graphite

Physical properties of graphite.

Properties	Graphite
Density (g/cm^3)	2.26
Young's Modulus (GPa)	8 - 15
Tensile Strength (MPa)	9 - 34
Compression Strength (MPa)	20 - 200
Thermal Conductivity (W/m.K)	25 - 470

APPENDIX B

SAMPLE CALCULATION OF VOLUME, MASS AND THE THEORETICAL DENSITY OF THE COMPOSITES

1. **Volume of pallet, V**

$\boxed{V = \Pi r^2 h}$

$V = \Pi (40/2)^2 (5)$

$V = \underline{6283.19 \text{ mm}^3 = 6.283 \text{ cm}^3}$

2. **Mass of Aluminium powder needed for pure aluminium pallet**

$\boxed{\rho = \text{mass, m / volume,V}}$

Mass of Aluminium = ρ * volume

Mass of Aluminium = 2.7 * 6.283

Mass of Aluminium = $\underline{16.946 \text{ g}}$

3. **2wt% of Graphite and 98wt% of Aluminium**

Volume of Aluminium, 0.98 * 6.283 = 6.157cm³

Volume of Graphite, 6.283 – 6.157 = 0.1257 cm³

Mass of 98wt% Aluminium = ρ * volume

= 2.7 * 6.157

= $\underline{16.624 \text{ g}}$

Mass of 5wt% Graphite = ρ * volume

= 2.26 * 0.0332

= $\underline{0.284 \text{ g}}$

For mixture, **ρ = total mass / volume**

= (16.624 + 0.284) / 6.283

= $\underline{2.646 \text{ g/cm}^3}$

∴ Thus, mixing of 2wt% of graphite and 98wt% of aluminium consist of 16.624 g of aluminium powder 0.284 g of graphite powder.

Same goes to the composition of 4wt%, 6wt% and 8wt%, the calculation will be based on the calculation of 2wt% as per example.

APPENDIX C

TABULATED RESULT OF ALL TESTING

1.0 Density, Diameter and Thickness Measurement

Properties of compacted samples.

No	Graphite wt%	Thickness (mm)	Diameter (mm)	Density (g/cm^3)
1.	0	4.88	39.90	2.59
2.	2	4.92	39.94	2.46
3.	4	4.94	40.00	2.26
4.	6	4.88	39.96	2.13
5.	8	4.96	39.96	2.08

2.0 Hardness Measurement

Hardness measurement result.

No	Graphite wt%	Hardness (HRB)					
		1	2	3	4	5	Average
1.	0	20.4	21.5	25.6	20.3	23.0	22.2
2.	2	34.3	33.8	34.7	33.4	35.8	34.4
3.	4	51.3	51.9	52.8	53.2	51.5	52.1
4.	6	65.7	58.4	65.2	59.7	63.2	62.4
5.	8	67.2	71.8	66.6	69.3	63.5	66.7

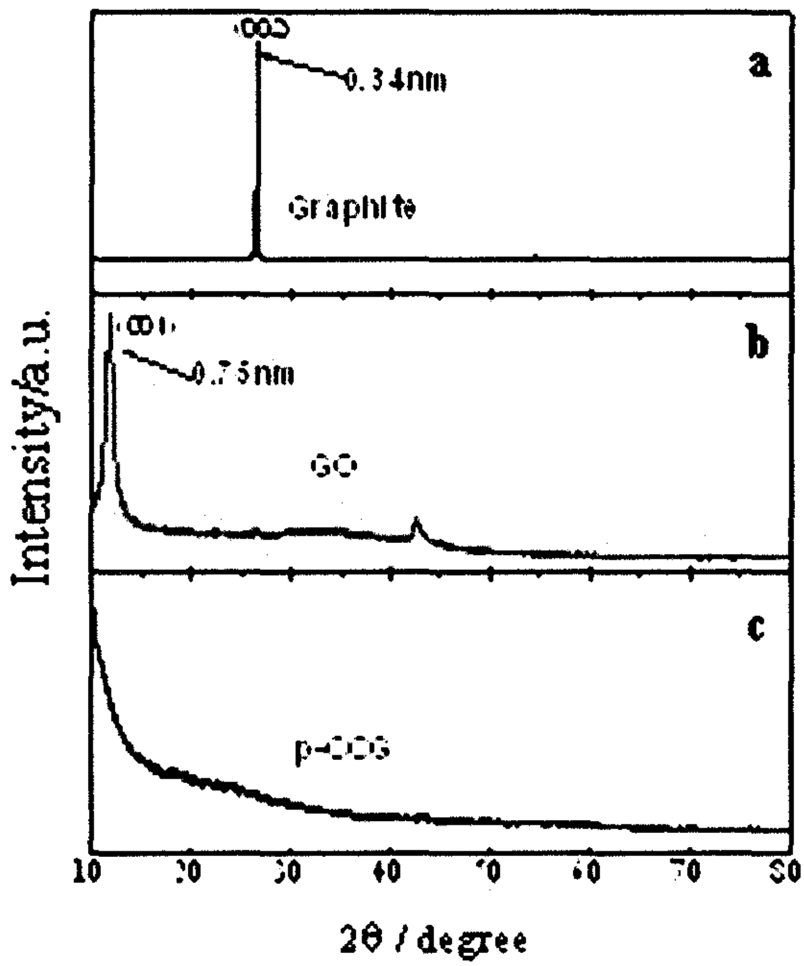
3.0 Wear Resistance Behavior

Wear resistance testing result.

No	Graphite wt% in Aluminium	% of Weight Loss		
		1 min	2 min	3 min
1.	0	0.34	0.46	0.56
2.	2	0.18	0.35	0.44
3.	4	0.25	0.38	0.47
4.	6	0.34	0.45	0.49
5.	8	0.35	0.48	0.59

APPENDIX D

XRD PATTERN OF PURE GRAPHITE



(a) Natural graphite (b) GO (c) p-CCG